

THE FRENCH REPUBLIC  
MINISTRY OF COMMERCE AND INDUSTRY  
PATENT AND TRADEMARK OFFICE  
**PATENT**

Group 14, Category 1    Number 769,586

**Method of Preparing Derivatives of Oxypyridine and Aminopyridine Combinations**

The company known as: *CHEMISCHE FABRIK VON HEYDEN Aktiengesellschaft*, which is registered in Germany.

**Application filed on March 5, 1934, at 1:05 P.M., in Paris.**

Issued on June 11, 1934. Published on August 28, 1934.

(Patent application filed in Germany on March 31, 1933. Applicant's statement.)

It has been observed that oxypyridine and aminopyridine combinations tend to combine as molecular combinations with substituted barbituric acids. Derivatives obtained in this manner form specific crystalline combinations whose physical properties essentially distinguish them from the raw materials which are used in each instance.

Derivatives of this kind are obtained from oxypyridine and aminopyridine combinations when the latter combinations are blended, for example, in order to yield an amount corresponding to 2 molecules for 1 or 2 molecules of a substituted barbituric acid. It is also possible, however, to cause the aforementioned components to act upon one another within solvents and to obtain molecular combinations by crystallization or evaporation, or to produce reactions between salts of barbituric acids and salts of the previously cited pyridine combinations.

*Example 1:* 2, 3 parts 4-oxy-5-nitropyridine and 3 parts diethylbarbituric acid

are dissolved in 60 parts of water according to volume, while being heated. As a result of cooling, a combination which melts at  $245^{\circ}$  is separated by crystallization, and, for 1 mole of  $C_8H_{12}O_3N_2$ , it contains 1 mole of  $C_5H_4O_3N_2$ .

*Example 2:* 1 part diethylbarbituric acid and 2 parts 2-ethoxy-5-acetaminopyridine are melted at  $140^{\circ}$ . After solidification of the clear melted mass, crystallization is obtained in water or benzene. The combination obtained in this manner melts at  $112^{\circ}$ - $115^{\circ}$ , and it contains 2 moles of  $C_9H_{12}O_2N_2$  for 1 mole of  $C_8H_{12}O_3N_2$ .

*Example 3:* 12 parts N-methyl- $\alpha$ -pyridone are added to a suspension consisting of 16 parts diethylbarbituric acid in 90 parts of heated benzene according to volume. As a result of stirring, a solution is promptly obtained. The combination, which is separated by crystallization during cooling, melts at approximately  $120^{\circ}$ , and it consists of the respective raw materials in a 1:1 ratio.

*Example 4:* 17 parts phenylethylbarbituric acid are intensively mixed

with 8 parts N-methyl- $\alpha$ -pyridone, and solidification occurs extremely quickly, with heat being released. After cooling, the product is powdered, and it is mixed with ether. Then it is filtered with a tube and is allowed to dry. In this way, it is possible to obtain a combination which melts at approximately  $122^{\circ}$  and contains 1 mole  $C_6H_7ON$  for 1 mole of  $C_{12}H_{12}O_3N_2$ .

*Example 5:* 20 parts diethylbarbituric acid and 25 parts  $\alpha$ -aminopyridine are dissolved in 100 parts of 20% alcohol by volume while being heated. After cooling, the crystals which are separated are filtered with a tube. Then rinsing with 20% alcohol and drying take place. It is possible to clean these crystals by dissolving them in ether and by precipitation with petroleum ether. The crystals then melt at  $82^{\circ}$ - $85^{\circ}$ . This combination contains 2 moles of  $C_5H_6N_2$  for 1 mole of  $C_8H_{12}O_3N_2$ .

## SUMMARY

The present invention pertains to:

1. A method of preparing derivatives of oxypyridine and aminopyridine combinations, characterized by the fact that said oxypyridine and aminopyridine combinations are caused to act upon substituted barbituric acids.
2. Methods of applying the process identified within 1, whereby:
  - a) The components are melted at an increased temperature.
  - b) The components are caused to act upon one another within a solution or a suspension.
  - c) A double reaction is produced between salts of the aforementioned components.

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